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The objective of this program was to carry out basic research on the phenomenon of surface-energy-driven secondary grain growth (SEDSGG) in thin films on amorphous substrates with surfaces which were planar as well as surfaces with artificial topography. Means of enhancing grain boundary mobility, such as ion bombardment, doping, and rapid thermal annealing were investigated. Theoretical models for SEDSGG were developed. The role of surface energy and surface topography in SEDSGG was characterized and understood.

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Final Report on Grant No.

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"Single Crystal Films of Semiconductors on Amorphous Substrates Via a Low Temperature Graphoepitaxy"

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Cambridge, MA 02139

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1.0 Introduction

The long-range motivation of this research effort was the development of a technology for producing single-crystal films on amorphous substrates. In response to suggestions from our sponsor in the latter half of the grant period, and because of some important phenomena we have observed (see Section 3.0), we emphasized the acquisition of basic understanding of the energetics and kinetics of grain growth in ultrathin films rather than developments aimed at short-term demonstrations of single-crystal films on amorphous substrates. By ultrathin films we mean films with thicknesses in the sub-500 Å range, in which surface energy driving forces play a significant role. Our efforts have recently expanded to also include investigations of ultrathin films on single-crystal substrates, in recognition of new insights we have gained on the possible role of surface-energy-driven grain growth in some types of heteroepitaxy. Until the initiation of our research program there were few enquiries into the role of surface energy anisotropy in morphological and structural changes. We are confident that the basic studies pursued under this grant will provide the underpinnings for achieving the long-range technological objective: new film configurations for advanced electronic systems.

In Sections 2-8 of this report we describe briefly the knowledge we have gained about grain growth using a variety of approaches, and summarize our current views in Section 9.

2.0 Suface-Energy-Driven Grain Growth - Basic Concepts

The phenomenon of surface-energy-driven grain growth is depicted schematically in Fig. 1. If a polycrystalline film is sufficiently thin, normal grain

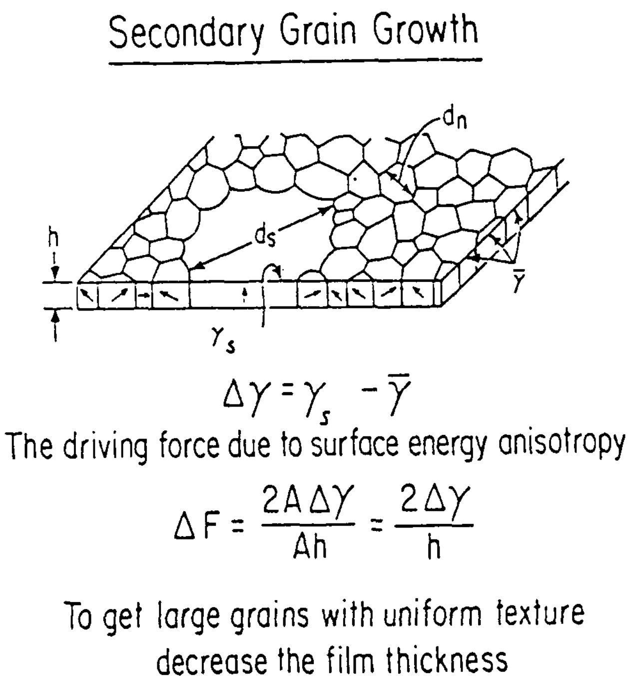

Figure 1. Schematic depiction of surface-energy-driven grain growth (SEDGG). A grain with minimum surface energy, γ_s, is shown growing into a matrix of normal grains with average surface energy $\bar{\gamma}$.

growth will tend to produce columnar grains in which the boundaries are perpendicular to the surface plane. Normal grain growth, which is driven by the reduction of grain boundary energy, occurs if the grain boundaries are able to move. However, normal grain growth tends to cease when the columnar grain diameters are approximately twice the film thickness. Among the columnar grains, some are oriented such that their surfaces have minimum energy. These grains can grow further to consume their neighbors, becoming large secondary grains with a specific crystallographic texture. This is the phenomenon we call surface-energy-driven secondary grain growth (SEDSGG). Our studies of this phenomenon have been the most extensive to date and have focused on thin films of semiconductors as well as metals. The driving force for SEDSGG includes a term due to surface energy minimization, as depicted in Fig. 1, and also a term due to grain boundary energy.

$$\Delta F \cong \frac{2\Delta\gamma}{h} + \frac{\gamma_{gb}}{h} \qquad (1)$$

In this equation, $\Delta\gamma$ is the difference between the surface energy of the growing secondary grain, γ_s, and the average surface energy of neighboring normal grains, $\bar{\gamma}$. The grain boundary energy is γ_{gb} and h is the film thickness. A much more detailed discussion of the theory of SEDSGG can be found in our publications. (J1-J8).

In our experimental and theoretical work to date we have focused on SEDSGG in films on amorphous substrates. If these substrates are planar, γ_s has a minimum value for grains with restricted textures, but there is no restriction on in-plane or azimuthal orientations. On patterned or single-crystal substrates, however, γ_s has minimum values for grains with 3-dimensionally constrained orientations. That is, secondary grains will differ in surface energy depending on their in-plane orientation relative to the single-crystal or patterned substrate. It is likely that in many cases of heteroepitaxy that occur by Volmer-Weber growth, orientation is achieved not at the stage where discrete islands exist on the substrate but at the stage where islands coalesce. In this case, the achievement of epitaxy should be considered a form of surface-energy-driven secondary grain growth. This viewpoint was not clearly expressed in the literature until the research conducted under this grant.

Figure 2 illustrates how surface-energy-driven secondary grain growth (SEDSGG), in conjunction with patterning of an amorphous substrate surface, can lead to a film with a specific in-plane orientation as well as a specific axis perpendicular to the film. Growth to impingement of three-dimensionally oriented grains can lead to single-crystal films.

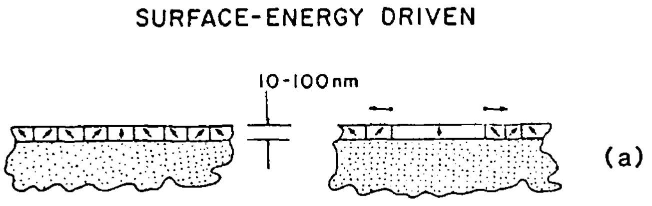

Figure 2(a). Schematic cross section of a film undergoing SEDSGG. The grain with minimum interfacial energy by virtue of its orientation grows by consuming grains with other orientations.

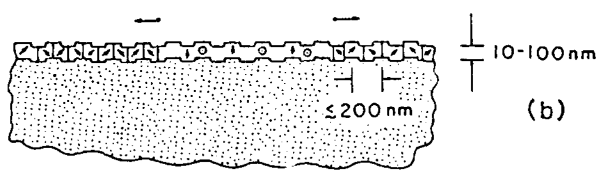

Figure 2(b). SEDGG in conjunction with surface patterning (solid-state-graphoepitaxy). A grain of minimum interfacial energy is one that is oriented relative to the surface pattern as well as the substrate normal.

The phenomena observed in thin and ultrathin films are more complex than depicted in Figs. 1 and 2. For example, the top surfaces of polycrystalline films are not planar as depicted. Instead, grooves form at grain boundaries. Some of the most startling phenomena we have observed were in films so thin (150Å) that they formed a network, but were not entirely continuous. Despite the discontinuous nature of the films, extremely large grains with a specific crystallographic texture were formed by SEDSGG. This is discussed in Section 3.

A major component of our efforts were on means of enhancing grain boundary mobility in films of Si and Ge. Such studies provided new information on how grain boundaries move in covalently bonded materials, as well as offering hope that SEDSGG can be applied at moderate or low temperatures. These studies are described in Sections 4 to 6.

3.0 SEDSGG in Ultrathin (≤150Å) Films

In ultrathin films of Ge, about 150 Å thick, secondary grains many micrometers in diameter were observed after annealing, despite the presence of a high density of voids in the film (J6). One would normally expect that such voids would pin grain boundary motion and thereby suppress formation of large secondary grains. We attribute these somewhat surprising results in part to very high surface-energy driving forces. Presumably, in such ultrathin films the driving

force is sufficiently high to overcome the inhibitory effects of the voids. The anomalous secondary grain growth in ultrathin Ge films is similar to secondary grain growth observed in 150 Å-thick films of Au by an earlier graduate student, Chee Wong (J3, C7).

4.0 Ion-Beam-Enhanced Grain Growth

Graduate student Harry Atwater conducted a broad study of the enhancement of grain boundary motion by means of ion bombardment (J7, J9, C8, C9, C14). His study of ion-beam-enhanced grain growth (IBEGG) was the first of its kind and has opened up new fields of study and engineering. He studied Ge, Si, and Au films less than 1000Å thick. Ion beams in the 40 - 100 keV range were employed, resulting in an ion damage profile whose peak was approximately in the middle of the thin film. Concurrent with ion bombardment, samples were annealed at 500 - 1000°C for Ge and Si, and at room temperature for Au. The temperature was chosen so that ion damage was annealed dynamically. IBEGG was characterized by varying the ion dose, ion energy, ion flux, ion species, temperature, and thin film deposition conditions. The effect of these parameters on grain size and microstructure was analyzed both qualitatively and quantitatively using transmission electron microscopy (TEM). A transition state model (J7, J9) was developed to describe the motion of grain boundaries during ion bombardment. The model accounts for the dependence of IBEGG on all experimental parameters. An atomistic picture of the jump rate at grain boundaries during IBEGG was proposed. Monte-Carlo simulation of ion range and defect production was performed using the TRIM code and a modified Kinchin Pease formula. The calculated defect yield per incident ion was correlated with enhanced grain growth, and used to estimate the number of atomic jumps at the grain boundary per defect generated at the boundary for a given driving force, a quantity which is approximately constant for a given film material. The IBEGG and thermal growth rates have been related to their respective point defect populations. That is, the grain growth rate appears to depend only on the concentration of vacancies and interstitials, irrespective of whether they are created thermally or by ion bombardment. We consider this an important finding.

5.0 The Effect of Dopants on Grain Growth in Silicon

In our studies of ion-beam-enhanced grain growth we arrived at the conclusion that grain boundary mobilities can be greatly enhanced by injection of point defects at the boundaries. We reached the same conclusion from separately funded studies of the effects of dopants on grain growth in polycrystalline silicon films.

We have observed that doping with electron donors, specifically phosphorous and arsenic, leads to significant enhancement of the rates of both normal and secondary grain growth in silicon films. We have also observed that doping with boron has little or no effect on either sort of grain growth. However, codoping with boron as well as with an acceptor leads to compensation of grain

boundary mobility enhancement. We carried out extensive studies of normal grain growth in silicon in order to develop an understanding of the Fermi energy dependence of grain boundary mobility.

We chose to study normal grain growth in order to avoid effects due to surfaces. This allows more direct determination of grain boundary mobilities. However, the mechanisms of grain boundary motion (but not the driving force) should be the same for both normal and secondary grain growth. Normal grain growth is driven by the reduction of grain boundary energies. Phosphorous and arsenic are known to segregate to silicon grain boundaries and therefore almost certainly reduce grain boundary energies. The effect of P and As on grain growth must therefore be specifically due to enhancement of grain boundary mobilities.

P, As, and B doping affect the rates of self diffusion and oxidation in similar ways to their effects on grain boundary mobility. In these cases, kinetic enhancement has been ascribed to changes in point defect concentrations. Similarly, we have related measured grain boundary mobilities to the total vacancy concentration. Vacancies in silicon can be neutral or have a single negative, double negative or single positive charge. While the concentration of neutral vacancies remains fixed, the concentration of negatively charged vacancies increases with increasing Fermi energy. While the concentration of positively charged vacancies increases with decreasing Fermi energy, the net effect on the total vacancy concentration is negligible. Because the energies for the charged vacancies are known, it is possible to calculate the total vacancy concentration as a function of electron concentration and temperature.

When we assume that grain boundary motion occurs through both a diffusive process (e.g. grain boundary dislocation climb) and a non-diffusive process (e.g. grain boundary dislocation glide), we find that the diffusive process scales with the total vacancy concentration. Calculations of grain growth rates predicted using this model are shown as lines and are accompanied by experimental points in Figure 3.

From these results we concluded that increased concentrations of point defects cause increases in grain boundary mobilities (C12,C13). In the case of ion-beam-enhanced grain growth, point defects are generated athermally due to atomic collision while here vacancies are generated due to the presence of dopants.

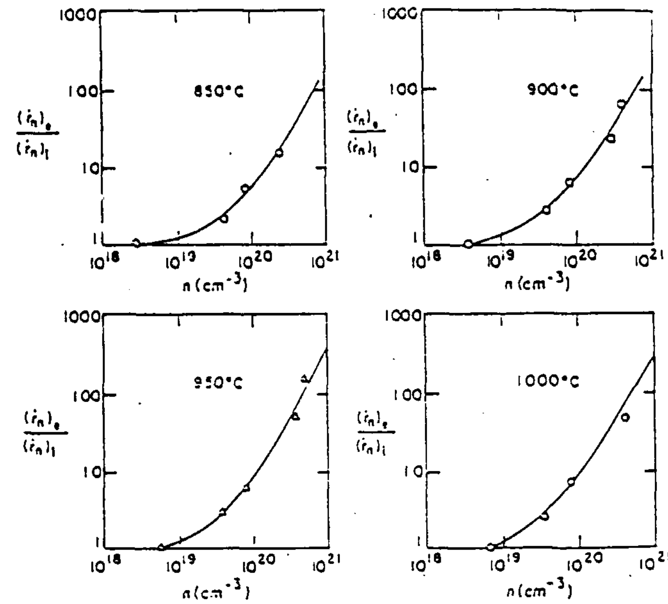

Figure 3. Ratios of the extrinsic and intrinsic normal grain growth rates as a function of electron concentration n and annealing temperature. Lines indicate theoretical predictions and points represent experimental results.

6. Grain Growth by Rapid Thermal Processing

We carried out a brief investigation of rapid thermal annealing (RTA) of doped polycrystalline silicon on amorphous SiO_2 in order to determine the time dependence of secondary grain growth at very short times (J5, C11). In the work described in section 5, we observed that the rate of secondary grain growth in doped polysilicon was not constant and in fact saturated at very short times. By carrying out RTA experiments we were able to establish that SEDSGG occurred at a constant rate only for the first few seconds of an anneal. During this time, however, the rate of growth was quite high (approximately 700Å/sec at 1100° C and 4500Å/sec at 1200° C). The origin of saturation of grain growth is not completely clear at this time. However, through cross-sectional electron microscopy we have observed grain boundary grooves, suggesting that saturation probably occurs due to groove formation.

Our experiments on rapid thermal annealing of polysilicon also yielded an unanticipated new insight. A number of groups have been investigating homoepitaxial transformation of polycrystalline silicon films deposited on single-crystal substrates. When these results are compared to our results we find virtually identical time, temperature and dopant concentration dependencies (C11). This comparison suggests that homoepitaxial transformation of polycrystalline silicon can also occur through a surface-energy-driven grain growth process. This experimental result, as well as theoretical analyses to be described below, encouraged us in our decision to investigate heteroepitaxy which occurs via grain boundary motion.

7.0 Basic Mechanisms of Heteroepitaxy

In the latter part of the grant period we focused attention on the issue that surface-energy-driven secondary grain growth can lead to conventional hetero-epitaxy as well as graphoepitaxy. In order to constrain 3-dimensionally the orientations of secondary grains, it is necessary that the surface energy be anisotropic

for rotations in the plane of the film. This anisotropy can be provided by artificial surface-relief structures (i.e., graphoepitaxy) or by a single crystal substrate.

We anticipate that in some cases epitaxy occurs despite the initial nucleation and growth of discrete, randomly oriented islands. In such a system, epitaxial alignment occurs when islands coalesce, at which point grain boundary motion and grain growth can occur. Epitaxy due to grain growth driven by surface energy minimization can be promoted by lattice matching but does not require lattice matching. In our review of the literature on epitaxy we have identified a number of "anomalies" which can be explained using the model outlined above.

8.0 Theoretical Developments

We have recently extended the theory of Lifshitz, Shyozov and Wagner (LSW) for three-dimensional particle coarsening to two-dimensional particle coarsening and grain growth in thin films (J8). We have specifically modified the theory to account for interface energy anisotropy, and therefore secondary grain growth. With this formal structure we will be able to quantitatively analyze the effects of the orientation dependence of the interface energy, e.g., as represented in a Wulff plot. We are now also able to predict the nature and evolution of distribu-ions of secondary grain sizes.

In addition to analytic modeling of secondary grain growth, under separate funding, we also developed computer models for microstructural evolution in thin films, including nucleation and growth to impingement, normal grain growth and secondary grain growth.

9.0 Current Perspectives on Surface-Energy-Driven Secondary Grain Growth

In thin films, especially ultrathin films, surface energy can have dominant importance in phase stabilization and in driving kinetic processes. In this program we were concerned with understanding and controlling microstructural evolution in thin films. From our work it is clear that surface-energy-driven grain growth is a process of great importance both during film formation and during subsequent processing. It can be the principal process controlling the final structure of films on planar or patterned amorphous substrates as well as on single crystal substrates.

Surface-energy-driven secondary grain growth can be controlled by modifying the driving force for growth or by modifying the grain boundary mobility. Competing processes such as grain boundary grooving (which is also surface-energy-driven) can impede grain growth due to energetic reasons but may in some cases promote orientation selectivity. High surface energy anisotropy also promotes selectivity. Orientation of secondary grains can be three-dimensionally constrained due to surface topography on amorphous substrates or due to interface energy minimization on single crystal substrates. In all cases, the total driving force for SEDSGG

increases with decreasing film thickness. This has been clearly confirmed in our experiments on germanium.

Grain boundary mobilities can be increased by generation of point defects. This has been demonstrated in experiments using ion bombardment and electronically active dopants in silicon.

10.0 Future Plans

The investigation of SEDSGG on single-crystal substrates and of the early stages of heteroepitaxy will be continued under a new grant through the efforts of a senior graduate student. Our progress in this area will be reviewed in our first annual report (April 1989).

11.0 Publications Under Grant

1. Journal Articles

J1 C.V. Thompson, "Secondary Grain Growth in Thin Films of Semiconductors: Theoretical Aspects," J. Appl. Phys. 58, 763 (1985).

J2 H.-J. Kim and C.V. Thompson, "Compensation of Grain Growth Enhancement in Doped Silicon Films," Appl. Phys. Lett. 48, 399 (1986).

J3 C.C. Wong, H.I. Smith, and C.V. Thompson "Surface-Energy-Driven Secondary Grain Growth in Thin Au Films," Appl. Phys. Lett. 48, 335 (1986).

J4 H.J. Frost and C.V. Thompson, "The Effect of Nucleation Conditions on the Topology and Geometry of Two-Dimensional Grain Structures," Acta Metallurgica 35, 529 (1987).

J5 S.M. Garrison, R.C. Cammarata, C.V. Thompson, and H.I. Smith, "Surface-Energy-Driven Grain Growth During Rapid Thermal Annealing (<10s) of Thin Silicon Films," J. Appl. Phys. 61, 1652 (1987).

J6 J. Palmer, C.V. Thompson, and H.I. Smith, "Grain Growth and Grain Size Distribution in Thin Germanium Films on SiO2," J. Appl. Phys. 62, 2492 (1987).

J7 H.A. Atwater, C.V. Thompson and H.I. Smith, "Interface Limited Grain Boundary Motion During Ion Bombardment," Phys. Rev. Lett. 60, 112, 1988.

J8 C.V. Thompson, "Coarsening of Particles on a Planar Substrate: Interface Energy Anisotropy and Application to Grain Growth in Thin Films," Acta Metallurgica 36, 2929 (1988).

J9 H.A. Atwater, C.V. Thompson and H.I. Smith, "Ion Bombardment Enhanced Grain Growth in Germanium, Silicon and Gold Thin Films," J. Appl. Phys. 64, 2337 (1988).

2. Published Conference Proceedings

C1 C.C. Wong, H.I. Smith, and C.V. Thompson, "Room Temperature Grain Growth in Thin Au Films," 4th International Symposium on Grain Boundary Structure and Related Phenomena, Supplement to Trans. of Jap. Inst. of Metals 27, 641 (1986).

C2 H.-J. Kim and C.V. Thompson, "The Effect of Dopants on Grain Boundary Mobility in Silicon," 4th International Symposium on Grain Boundary Structure and Related Phenomena," Japan Institute of Metals, Supplement to Trans. of Jap. Inst. of Metals 27, 495 (1986).

C3 H.J. Kim and C.V. Thompson, "The Effects of Dopants on Surface-Energy-Driven Grain Growth in Ultrathin Si Films," Proc. fall meeting of the Mat. Res. Soc., Boston, MA, Dec. 2-6, (1985), Mat. Res. Soc. Symp. Proc. 54, 730 (1986).

C4 C.V. Thompson and H.I. Smith, "Secondary Grain Growth in Thin Films," Phase Transitions in Condensed Systems - Experiment and Theory, Mat. Res. Soc. Symp. Proc. 57, 499 (1987). eds. G.S. Cargill III, F. Spaepen K.N. Tu, The Materials Research Society.

C5 H.J. Frost and C.V. Thompson, "Modelling of Thin Film Grain Structures and Grain Growth," Proceedings of Computer Based Microscopic Description of the Structure and Properties of Materials Symposium of the fall meeting of the Materials Research Society, December (1985).

C6 H.J. Frost and C.V. Thompson, "Modelling of Thin Film Grain Structures and Grain Growth," Proceedings of the Computer Based Microscopic Description of the Structure and Properties of Materials Symposium, Proceedings of the 1985 fall meeting of the Materials Research Society, Boston, MA 1985.

C7 C.C. Wong, H.I. Smith, and C.V. Thompson, "Secondary Grain Growth and Graphoepitaxy in Thin Au Films on Submicrometer-Period Gratings," Mat. Res. Soc. Symp. Proc., 47, 35 (1985), Materials Research Society.

C8 H.A. Atwater, H.I. Smith, and C.V. Thompson "Enhancement of Grain Growth in Ultra Thin Germanium Films by Ion Bombardment," Mat. Res. Soc. Symp. Proc., 51, 337 (1986), eds. H. Kurz, G.L. Olson, J.M. Poate, the Materials Research Society.

C9 H.A. Atwater, H.I. Smith, and C.V. Thompson, "Ion Beam Enhanced Grain Growth in Thin Films," Beam-Solid Interactions and Transient Processes Symposium, Materials Research Society Meeting, Dec., 1986. Mat. Res. Soc. Symp. Proc. 74, 499 (1987).

C10 H.I. Smith, M.W. Geis, C.V. Thompson, and C.K. Chen, "Crystalline Films on Amorphous Substrates by Zone Melting and Surface-Energy-Driven Grain Growth in Conjunction with Patterning," Mat. Res. Soc. Symp. Proc., 53, 3 (1986). eds. A. Chiang, M.W. Geis, L. Pfeiffer, the Materials Research Society.

C11 R.C. Cammarata, C.V. Thompson, and S.M. Garrison, "Secondary Grain Growth During Rapid Thermal Annealing of Doped Polysilicon Films," presented at Spring MRS meeting, 1987, Mat. Res. Soc. Symp. Proc. 92, 335 (1987).

C12 C.V. Thompson, "Dopant and Ion Beam Enhanced Grain Growth in Polycrystalline Silicon Films (Invited), accepted for publication in "Diffusion Processes in High Technology Materials," ed. by D. Gupta and A.D. Romig, Jr., Symposium of TMS/AIME fall meeting, Cincinnati, OH, 1987.

C13 C.V. Thompson, "Grain Growth in Polycrystalline Silicon Films" (Invited), accepted for publication in the proceedings of the Fall 1987 Materials Research Society Symposium on Polysilicon Films and Interfaces, Boston, MA.

C14 H.A. Atwater, C.V. Thompson, and H.I. Smith, "Transition State Model for Grain Boundary Motion During Ion Bombardment" (Invited), accepted for publication in the proceedings of the Fall 1987 Materials Research Society Symposium on the Fundamentals of Beam-Solid Interactions and Transient Thermal Processing, Boston, MA.

C15 C.V. Thompson, "Observations of Grain Growth in Thin Films" (Invited), to appear in the proceedings of the Topical Symposium on Microstructural Science for Thin Film Metallization in Electronic Applications, Spring Meeting of the Metallurgical Society of the American Institute of Mining, Metallurgical and Petroleum Engineers, Phoenix, AZ, Jan. 1988.

3. Theses

J.E. P[illegible]ner, "Secondary Grain Growth in Ultra Thin Germanium Films on Silicon Dioxide," M.S. Thesis, Department of Electrical Engineering and Computer Science, Massachusetts Institute of Technology, August, 1985.

C.C. Wong, "Secondary Grain Growth and Graphoepitaxy in Thin Au Films," Ph.D. Thesis, Department of Materials Science and Engineering, Massachusetts Institute of Technology, February 1986.

S.M. Garrison, "The Kinetics of Secondary Grain Growth In Rapidly Thermal Annealed Thin Silicon Films," S.M. Thesis, Department of Materials Science and Engineering, Massachusetts Institute of Technology, June 1986.

H.A. Atwater, "Ion Beam Enhanced Grain Growth in Thin Films," Ph.D. Thesis, Department of Electrical Engineering and Computer Science, Massachusetts Institute of Technology, June, 1987.